

## Preparation of novel Schiff base complexes of some transition metal (II) derived from 4,4'-ethylenedianiline and antibacterial assessment

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### Abstract

A particular number of complexes, which include Pd (II), Mn (II), Zn (II), and Ag (I) ions, established from the Schiff base ligand 4,4'-(ethane-1,2-diyl)bis(N-(pyridin-2-ylmethylene)aniline) ( $C_{26}H_{22}N_4$ ) as a 1:1 Molar ratio, were synthesized. We prepared the Schiff base by condensing 4,4'-ethylenedianiline and pyridine-2-carboxaldehyde. and characterized the Schiff base ligand and metal complexes using elemental analysis, melting point determination, molar conductance measurement, FT-IR spectroscopy, <sup>1</sup>HNMR spectroscopy, powder X-ray diffraction, and biological effectiveness evaluation from the infrared spectrum, electronic spectra, and <sup>1</sup>HNMR data. The analysis revealed that the Schiff base ligand donates the electrons of nitrogen (N) atoms to the metal ions. The suggested general formula for the obtained complexes is  $[M(L)]Cl_n$  for Pd (II) and Ag (I) metal ions n can be either 1 or 2. and The formul

$[M(L)(H_2O)_2]OAc_2$  for Mn(II) and Zn(II) metal ions (L) is a Schiff base ligand with the chemical formula  $C_{26}H_{22}N_4$ . The proposal suggests the existence of a four-coordinated square planar geometry for the Pd(II) and Ag(I) complexes and a six-coordinated octahedral geometry for the Mn(II) and Zn(II) complexes. The molar conductance measurements of all the complexes indicated their electrolytic character. The silver complex has a conductivity as 1:1 ratio and 2:1 for other complexes. In a laboratory setting, we assessed the antibacterial properties of the Schiff base ligand and its metal complexes against Gram-positive bacteria (*Staphylococcus typhi*) and Gram-negative bacteria (*Escherichia coli* and *Pseudomonas aeruginosa*) using the Agar disc diffusion method. The antibacterial testing findings demonstrated that the metal complexes displayed a higher level of antibacterial efficacy compared to the unbound Schiff base ligand.

**Keywords:** Schiff base, Mn ,Zn, Pd (II) complexes, anti bactericidal

تحضير معقدات قواعد شيف الجديدة لبعض العناصر الانتقالية (II) المشتقة من 4, -4 -

أثيلين ثنائي أنيلين وتقييم الفعالية المضادة للبكتيريا

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## الخلاصة

تم تحضير معقدات جديدة للمغنيز والخاصين والبلاديوم الثنائية والفضة الاحادية لليكاند قاعدة شيف

4,4-(ethane-1,2-diyl)bis(N-(pyridin-2-ylmethylene)aniline) (C<sub>26</sub>H<sub>22</sub>N<sub>4</sub>),

حيث تم تحضير قاعدة شيف بتفاعل تكثيفي بين البريديين 2- كاربوكسالديهايد و المركب 4,4- أثيلين ثنائي الانيلين تم تشخيص الليكاند والمعقدات الناتجة بعدة تقنيات منها تحليل العناصر وطيف الاشعة تحت الحمراء وطيف الرنين النووي المغناطيسي للبروتون , وقياس حيود الاشعة السينية XRD والتوصيلية المولارية وقياس درجة الانصهار كونت المعقدات المحضرة ومن النتائج المستحصلة الصيغ التركيبية الاتية  $[M(L)(H_2O)_2] OAc_2$  بالنسبة لمعقدات المغنيز والخاصين والصيغة

$[M(L)] Cl_n$  لمعقدات البلاديوم والفضة . كما اثبتت قياسات طيف الاشعة تحت الحمراء وطيف الرنين النووي المغناطيسي للبروتون ان الليكاند يتناسق من خلال ذرتي نيتروجين مجموعة الازوميثين لقاعدة شيف ونيتروجين الحلقة البريدينية مع الايونات الفلزية مكوناً معقدات للمغنيز والخاصين سداسية التناسق ذات شكلا هندسيا ثماني السطوح بينما كونت معقدات البلاديوم والفضة رباعية التناسق الشكل الهندسي مربعا مستويا كما بينت قياسات التوصيلية المولارية ان المعقدات موصلة بنسبة 1:2 وبالنسبة لمعقد الفضة 1:1 ، تم اختبار ليكاند قاعدة شيف ومعقداته كمضادات للبكتيريا باستخدام تقنية الانتشار على سطح الاكار باستخدام أنواع من البكتيريا السالبة الغرام مثل البكتيريا الاشريكية والسيدوموناس اوراجينوزا واصناف من البكتيريا الموجبة الغرام مثل الستافيلوكوكس تايفي حيث بينت النتائج ان المعقدات المحضرة اكثر فعالية في قتل البكتيريا قاعدة شيف المطلقة كليكاند .

**الكلمات المفتاحية:** قواعد شيف ، معقدات المغنيز والخاصين والبلاديوم ، مضادات البكتيريا

## 1. Introduction

The scientific community continues to be intrigued by the Schiff base ligands and their metal complexes, which have nitrogen as a donor atom. The coordination of these ligands with the metal atom is highly facile. The ligands are produced through a classical method involving primary amines and aldehydes. Depending on the specific reaction conditions ,these ligands exhibit varying coordination modes with the metal atom. [1]

The major objective of the research is to develop reliable and efficacious pharmacological medications for managing the symptoms caused by bacteria and cancer. a significant area of concentration pertains to the biological activities of Schiff base metal complexes. Various Schiff base metal complexes exhibit various biological and pharmacological impacts. The medical significance of Coordination compounds formed by combining metal ions with Schiff base ligands containing "N" donor atoms lies in their antibacterial, antifungal, and anti-inflammatory activities. [2], analgesic [3, 4], anti-diabetic [5], antitubercular [6], antioxidant [7] [8]. The complexes of Schiff have been employed as biological drugs to understand the assembly of biomolecules and their physiological roles. [9] Manganese, zinc, palladium, & Silver are essential elements for sustaining life. They have higher levels of biological activity when they are a part of particular metal protein complexes that aid in the transfer of electrons, the transport of oxygen, or the storage of ions. [10]. This has resulted in a surge of enthusiasm for investigating systems incorporating these metals. This article presents the results of a study that examined the antibacterial properties of newly developed complexes of Mn (II), Pd (II), Zn(II), and Ag (I) with a Schiff base ligand entitled 4,4-(ethane-1,2-diyl)bis(N-(pyridin-2-ylmethylene)aniline) (C<sub>26</sub>H<sub>22</sub>N<sub>4</sub>), The findings of the anti-bacterial evaluation demonstrated that the metal complexes exhibited improved antibacterial effectiveness compared to the unbound Schiff base ligand against both Gram-positive and Gram-negative bacteria (*B. subtilis* and *S. typhi*), as well as *E. coli* and *P. aeruginosa*.

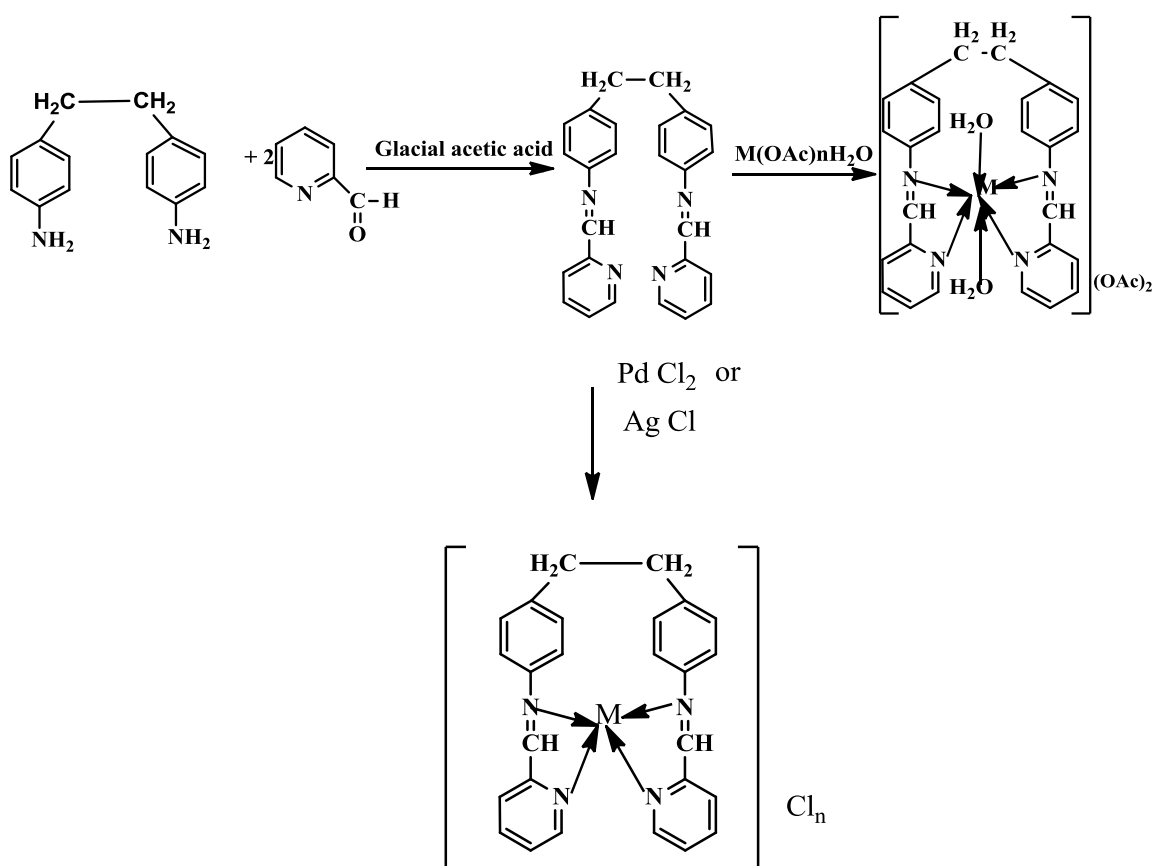
## 2. Experimental Part

The analytical grade chemicals and solvents utilized in this investigation were obtained from Sigma Chemicals Company. Standard literature procedures were utilized to synthesize Schiff base ligands in a pure solution. [11], The melting points were unadjusted and were determined using a Gallenkamp apparatus. The IR Spectroscopy of synthetic ligands and their complexes were measured using a SHIMADZU FTIR-8400 spectrophotometer. The spectra were taken within the region of 4000–400 cm<sup>-1</sup>. The <sup>1</sup>HNMR spectra of the Schiff base ligand were obtained using a Bruker Avance 400 MHz <sup>1</sup>HNMR spectrometer at room temperature. The measurements were conducted in deuterated DMSO. The internal standard used was tetramethylsilane. and there occurs a chemical shift in. A LECO CNHS-932 microanalyzer was used to record the elemental analysis (C, H, and N%). At room temperature, the conductivity of the Schiff base ligand and its metal (II) complexes was measured using an SIBATA Conductivitymeter Model SC-17A. The antibacterial activity of the accomplished compounds against *S. typhi*, *P. aeruginosa*, and *E. coli* has been tested.[12].

## 2.1. Preparation of ligand

With a few adjustments, the Schiff base ligand (L) was synthesized by published protocols [11].

4,4'-ethyldianiline (4.92 g; 0.023 mmol) was added at a 1: 2 molar ratios to a round-bottomed flask has a volume of 50 mL. of 2- pyridincarboxaldehyde (5 g; 0.046 mmol) in a magnetically agitated ethanolic solution. After adding few drops of acetic acid to the liquid until bring its PH down to around 6, a viscous yellow-orange precipitate quickly developed. Filtration was used to isolate the precipitate, and recrystallization from ethanol was used to purify it. To get rid of unreacted amine and aldehyde, the crystals performed a process of purification using diethyl ether. The air was employed to desiccate the crystals. 90.0% yield; color: yellow, m.p. = 144–145°C, C<sub>26</sub>H<sub>22</sub>N<sub>4</sub> (FW = 390) (Scheme 1 )



M= Zn, Mn, Pd, Ag

n= 1 or 2

(Scheme 1)

## 2.2. metal (II) Complex Synthesis

The complexes (Scheme 1) were synthesized by reacting the Schiff base with the metal ions using the protocols described in the literature. A mixture of ligand (L) (0.1 mmol) and 10 mL of hot ethanol at 60°C was gradually combined with a solution of hydrated metal salt (0.1 mmol) in 10 mL of water. Subsequently, the liquid underwent reflux for three hours, accompanied by magnetic stirring. The resultant product was subjected to filtration to remove any residual unreacted Schiff base and metal salt and subsequently rinsed with distilled water and diethyl ether. (Scheme 1) [11].

## 3. Assay for Biologicals

Using the disc diffusion method, The synthesized ligand and its transition metal complexes were assessed in a laboratory setting to determine their effectiveness in inhibiting the growth of bacteria strains, including *Escherichia coli*, *Pseudomonas aeruginosa*, *Bacillus subtilis*, and *Salmonella typhi*. [11, 13]. the method of disc diffusion was chosen because to its affordability, effectiveness, and ease of usage. The ligand and its metal complexes were produced as stock solutions in a solvent of dimethyl sulfoxide (DMSO). Concentrations of 500, 250, and 125  $\mu\text{g mL}^{-1}$  were achieved by diluting the stock solution, which acted as the control for each dilution. The bacteria's cultures were nourished using agar as the nutritional medium. Gentamicin and Amoxyclav, commonly used antibacterial drugs, were employed as positive controls alongside the metal complexes and ligands created. *E. Coli*, *P. aeruginosa*, and *S. typhi* were individually placed on Petri dishes containing nutrient agar medium. The dishes were then incubated for 24 hours to allow for growth. The average  $\pm$  standard deviation of three repeated measurements was utilized to compute the antibacterial efficacy (Table 3). The zones of inhibition (Figure 5) were measured in millimeters (mm) using a Vernier measurement instrument, employing the given criteria. [12].

## 4. Result and discussion

Feng [13,14] initially created the Schiff base ligand used in this investigation using the reflux technique. However, this study obtained a greater yield by preparing the ligand utilizing the room temperature approach. Since the ligand's metal complexes are novel, they are being reported for the first time. ethanol, DMF and DMSO can dissolve the Schiff base ligand. The colorful solids known as imines and their metal complexes are stable in air. dissolved in DMF and DMSO. Since the complexes' melting points are greater than the ligand's Schiff base, the complexes are thought to be more stable (Scheme 1) and display the reaction of prepared ligands and complexes.

## 5. Conductivity measurement

The molar conductance values of the generated complexes were measured in  $10^{-3}M$  Concentration of DMSO at a constant laboratory temperature. The electrical conductance values of the produced substances. were determined to be in a range  $80-88 \text{ Ohm}^{-1}\cdot\text{cm}^2\cdot\text{mol}^{-1}$ , suggesting their propensity to conduct electricity. 1: 1 The Ag complex was accepted as it exhibited electrolytic behavior .and 2:1 ratio For The Mn, Zn& Pd complex. [15, 16]. This implied the presence of  $(\text{OAc})_2$  and  $\text{Cl}_2$  anions outside the complexes' coordination region.

## 6. Elemental analysis

All of the complexes, according to the microanalysis results, appeared to be mononuclear, with one mole of prepared ligand and tow mole of water molecules coordinated to the central metal ion. As a result, the findings revealed that the complex's metal to ligand ratio was 1:1 and that the formula of complexes' was  $[\text{M}(\text{L}) (\text{H}_2\text{O})_2] (\text{OAc})_2$  ( $\text{M} = \text{Mn} (\text{II}), \text{Zn} (\text{II}),$  and  $[\text{M}(\text{L})\text{Cl}_n]$  for Pd (II) and Ag (I) complexes  $n= 1$  or  $2$ , [31, 32]. It was discovered that there was good agreement between the experimental and theoretical (calculated) results.

**Table 1: The physical properties and element analysis**

No.	m.p	Color	Yield %	Oh m	Mwt	C%cal(exp)	H% cal(exp)	N% cal(exp)
L	144-145	Yellow	90	4	390	80.00(79.73)	5.64 (5.59)	14.35(14.50)
1	343	light yellow	70	80	563	63.94(64.03)	4.97(5.15)	9.94(9.88)
2	362	light yellow	66	85	573.39	62.78(62.21)	4.88( 4.69)	9.76(9.65)
3	357	Yellow	73	88	614.42	58.59(59.01)	4.55(4.63)	9.25(9.11)
4	358	Yellow	70	44	533.5	58.48(59.07 )	4.12 ( 3.99)	10.49 10.23)

## 7. FT-IR spectroscopy

The binding mechanism of the Schiff base ligand to the metal ions in complexes was ascertained by comparing the FT-IR spectrum of the unbound ligand with the spectra of the metal (II) complexes. For the free ligand, The vibration frequencies of the azomethine C=N bond,  $\nu(\text{C}=\text{N})$ , was  $1651.0 \text{ cm}^{-1}$  [17-20]. The frequencies of C=N stretching in the metal (II) complexes were found to move to lower wave numbers [21-26] at 1568.0, 1564.0, 1556.0, and  $1555.0 \text{ cm}^{-1}$  for the complexes of manganese (II), zinc (II), palladium (II) and Silver (I), respectively. This demonstrated the Schiff base's interaction with the azomethine nitrogen. [27]. strong band is observed at  $1352.0 \nu(\text{C-N}_{\text{Py}})$  The stretching frequency of the C-N Py bonds in the complexes of metal ion (II) were observed at 1260.0, 1254.0, 1246.0 and  $1244.0 \text{ cm}^{-1}$  for Manganese (II), Zinc (II), palladium (II) and Ag (I), complexes, respectively This indicated coordination of pyridine nitrogen and metal ion.

the appearance of additional weak bands in the region  $416\text{--}453 \text{ cm}^{-1}$ ,  $406\text{--}436 \text{ cm}^{-1}$ ,  $406\text{--}442 \text{ cm}^{-1}$  and  $439\text{--}451 \text{ cm}^{-1}$  for complexes of Manganese (II), Zinc (II), palladium (II), and Silver (I), respectively attributed to  $\nu(\text{M-N})$  [28], further confirmed complexation [29]. Strong bands in the  $3331\text{--}3375 \text{ cm}^{-1}$  area were also visible in the complexes' FT-IR spectra, indicating the presence of coordinated/lattice water inside them. The nonligand band that showed in the  $693\text{--}696 \text{ cm}^{-1}$  region and was linked to the water's rocking progress further supported this. [29]

**Table 2: The most important bands in Infrared spectroscopy**

No.	Molecular structure	C=N	C-N Py	M-N	H <sub>2</sub> O-M	Cl-M
<b>L</b>	$\text{C}_{26}\text{H}_{22}\text{N}_4$	1651	1352	-	-	-
<b>1</b>	$[\text{Mn}(\text{L})](\text{OAc})_2$	1568	1260	416-453	696	-
<b>2</b>	$[\text{Zn}(\text{L})](\text{OAc})_2$	1564	1254	406-436	695	-
<b>3</b>	$[\text{Pd}(\text{L})]\text{Cl}_2$	1556	1246	406-442	-	527-580
<b>4</b>	$[\text{Ag}(\text{L})]\text{Cl}$	1555.0	1244	439-451	-	529-580

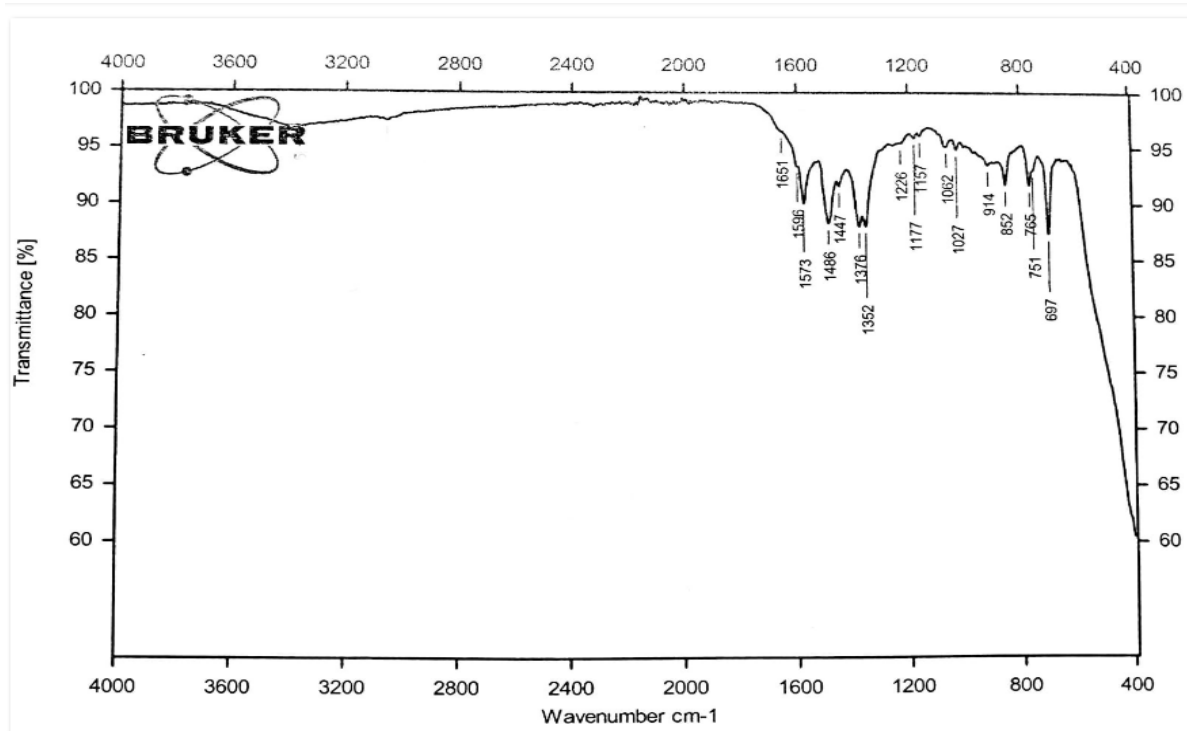


Fig. 1: FT-IR of Schiff base ligand L= C<sub>26</sub>H<sub>22</sub>N<sub>4</sub>

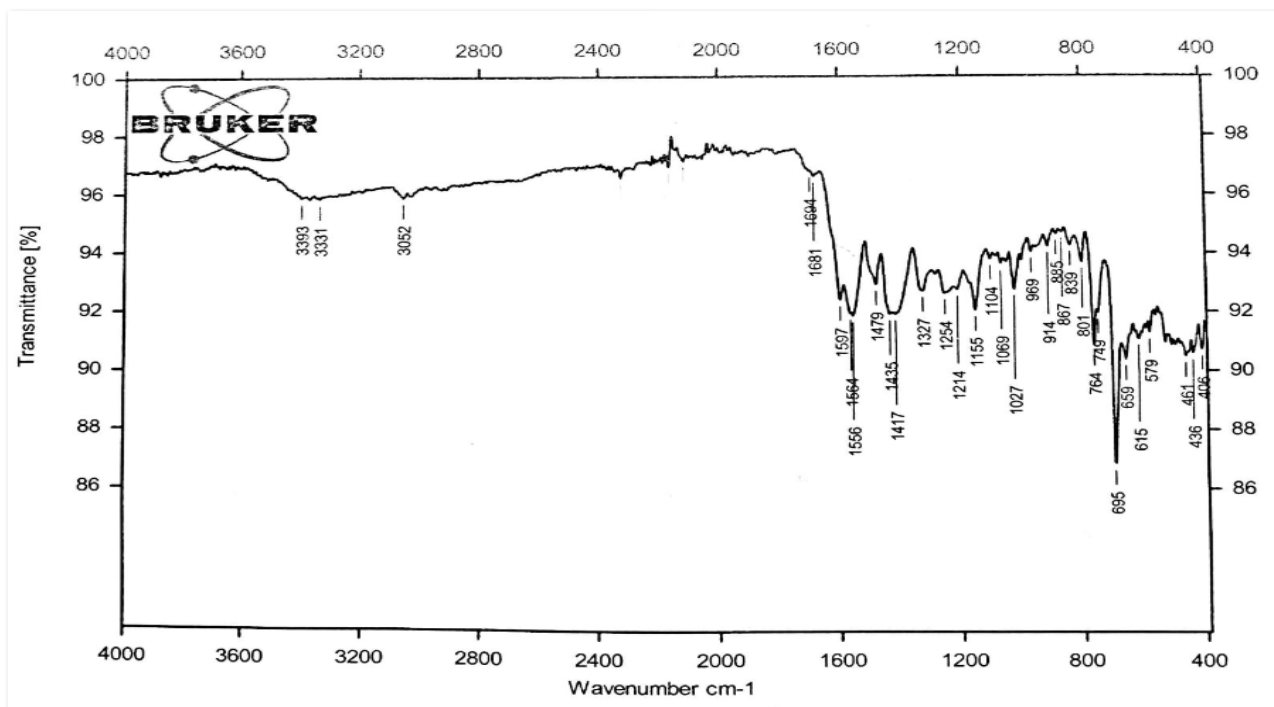


Fig. 2: FT-IR of [Zn ( L ) ( H<sub>2</sub>O )<sub>2</sub>] 2OAc



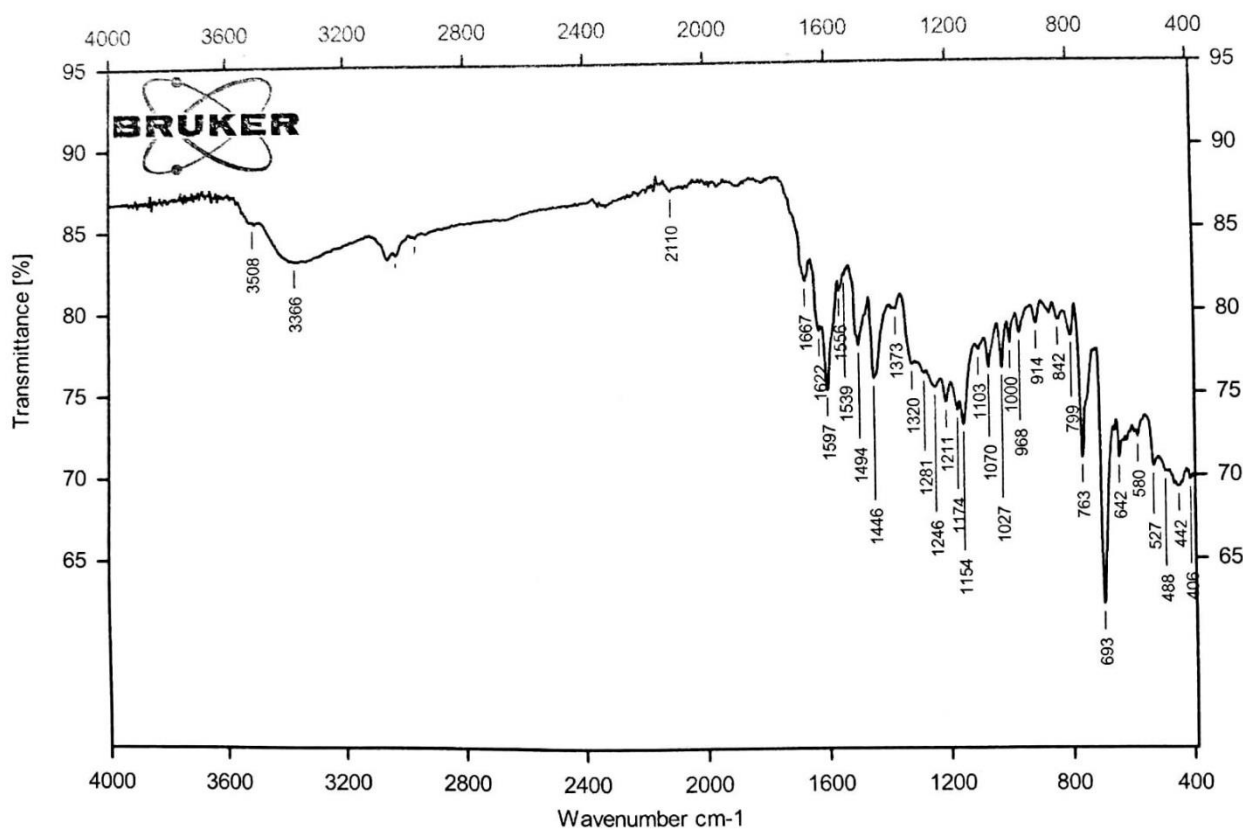


Fig. 3: FT-IR of [Pd ( L) ] Cl<sub>2</sub>

## 8. <sup>1</sup>H-NMR

The <sup>1</sup>H NMR spectra of ligand were recorded in DMSO-*d*<sub>6</sub>. The <sup>1</sup>H NMR spectrum of the Schiff base showed a singlet peak at  $\delta = 8.59$  ppm corresponding to the azomethine proton ( $-\text{N}=\text{CH}-$ ) [30, 31,33-37], an indication that the Schiff base was formed during the condensation reaction, multi peak at  $\delta = 7.19- 8.08$  ppm belong to protons of aromatic ring. peak at  $\delta = 2.89$  ppm belong to proton of CH<sub>2</sub>-CH<sub>2</sub> methyl group.

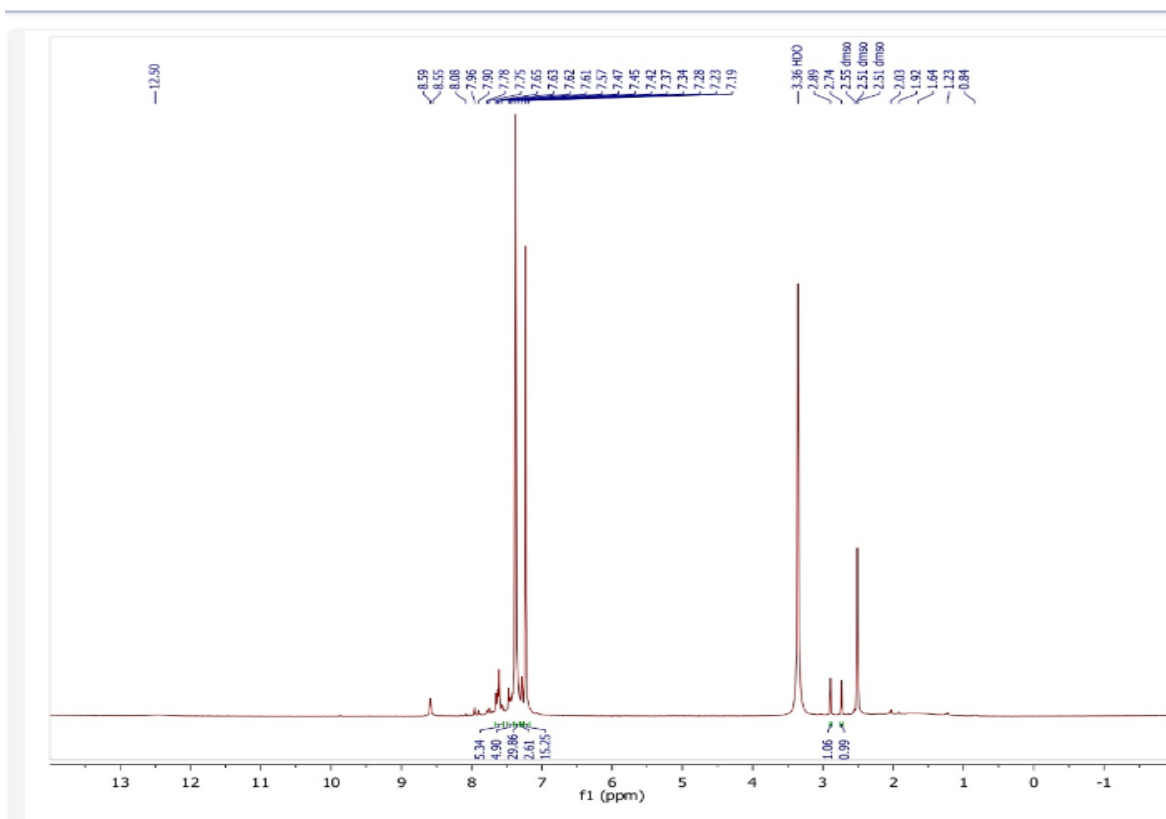


Fig. 4:  $^1\text{H}$ NMR of Schiff base ligand  $\text{L} = \text{C}_{26}\text{H}_{22}\text{N}_4$

## 9. Powder XRD of ligand

When measuring X-ray diffraction of the ligand, signals with sharp peaks were obtained indicating the presence of crystals in the sample. Scientifically X-ray diffraction is estimated the percentage of reflection of sample, as the peak width is inversely proportional to the crystallization process.

The XRD pattern of Schiff base were shown in Fig. 5 the peaks at  $12.9142^\circ$ ,  $16.5555^\circ$ ,  $17.7609^\circ$ ,  $19.7861^\circ$ ,  $21.2714^\circ$ ,  $23.2689^\circ$ ,  $24.0088^\circ$ ,  $24.9950^\circ$ ,  $25.9060^\circ$ ,  $29.8330^\circ$ ,  $31.8328^\circ$  and  $33.5143^\circ$  suggested that is polycrystalline structure. The 100% intensity appears at  $21.2714^\circ$  for Schiff base ligand indicate that ligand formation. The crystallite size were calculated for prominent peaks for the prepared Schiff base using Debye-Scherrer's formula [38]

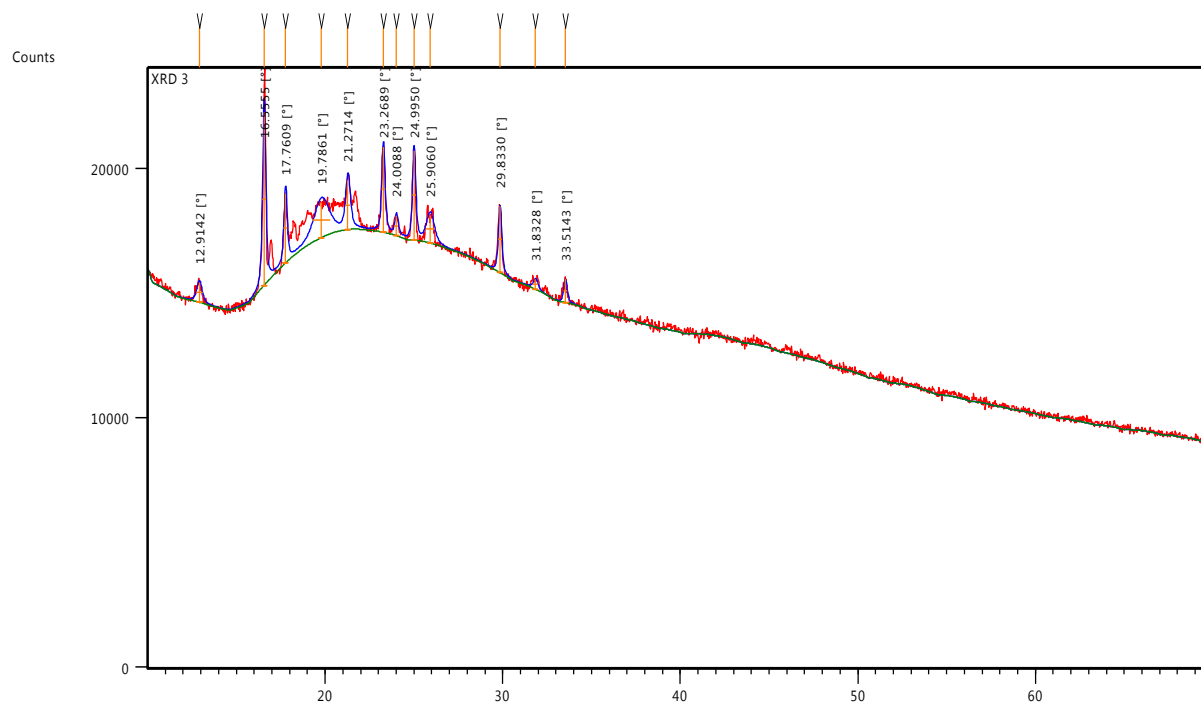
Fig. 5: XRD of Schiff base ligand L= C<sub>26</sub>H<sub>22</sub>N<sub>4</sub>

Table 3: The Peak list of XRD of the ligand

Pos. [°2θ]	Height [cts]	FWHM Left [°2θ]	d-spacing [Å]	Rel. Int. [%]	Tip Width
12.9142	784.53	0.3582	6.85528	11.28	0.4298
16.5555	6952.74	0.2047	5.35477	100.00	0.2456
17.7609	2800.53	0.2047	4.99398	40.28	0.2456
19.7861	1428.88	1.0234	4.48715	20.55	1.2280
21.2714	1994.58	0.3070	4.17708	28.69	0.3684
23.2689	3429.76	0.2047	3.82282	49.33	0.2456
24.0088	799.15	0.2047	3.70666	11.49	0.2456
24.9950	3585.67	0.2047	3.56261	51.57	0.2456
25.9060	1114.66	0.5117	3.43936	16.03	0.6140
29.8330	2683.67	0.2047	2.99497	38.60	0.2456
31.8328	401.36	0.3070	2.81123	5.77	0.3684
33.5143	956.52	0.2047	2.67393	13.76	0.2456

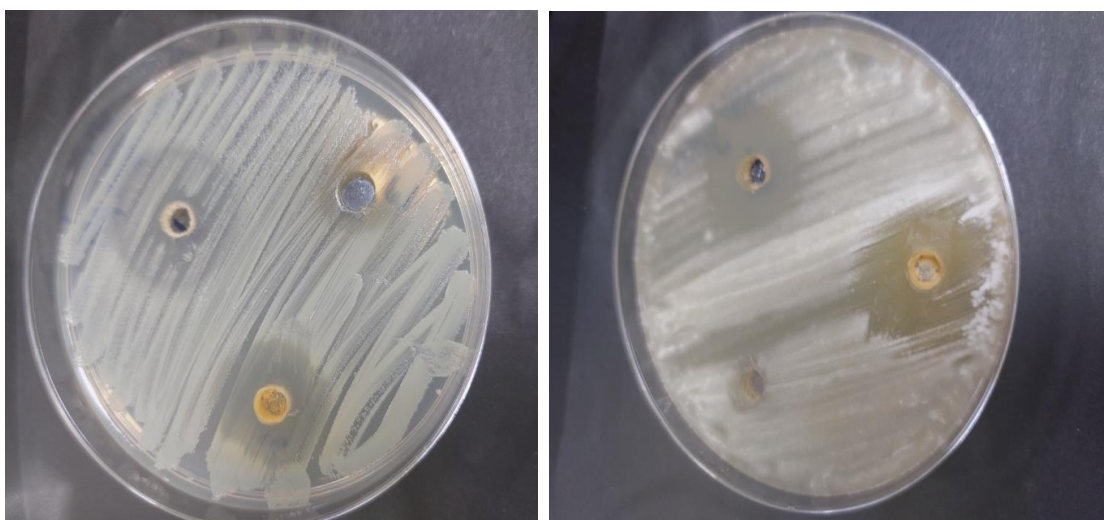
## 10. Antibacterial activity

The antibacterial activity testing on two Gram-positive strains and two Gram-negative bacteria strains was conducted according to established methods. The numbers are [13, 39]. The results were documented and displayed in Table 3. The efficacy of the well-established traditional antibacterial drugs, Amoxiclav (AMC) and Gentamicin (GEN) was compared to that of the ligand and its metal complexes in inhibiting bacterial growth. Evidence has shown that the ligand and its complexes can potentially hinder Bacterial growth. Studies have demonstrated that the coordination complexes can hinder growth of bacteria. results in the production of an antibacterial capacity to inhibit bacterial development. The number 40 is represented as [40]. Overall, the study found that the bactericidal activity of the complexes formed by the Schiff base ligand was stronger than that of the ligand itself, as indicated by the inhibitory zones observed at different concentrations. [41] Additionally, Amoxyclav (AMC), a common medicine, was tested against each Microbial variant. The concentration of metal complexes directly correlates with their antibacterial action. Multiple concepts have been proposed to clarify the biological mechanism of transition metal coordination complexes. The presence of metal ions in the bacterial cell process may be responsible for the heightened antibacterial activity observed in metal (II) complexes. The number [42]. Research has shown that enzymes can be deactivated by coordination with metals through the presence of structural components containing additional nitrogen donor atom systems, specifically (C=N) and (C-N py). The numbers [43, 44] Tweedy's chelation theory posits that the ligand orbital overlapping and partial sharing of positive charge between the metal ion and donor groups effectively reduce the polarity of the metal ion. [45, 46]. Hence, it is probable that these complexes might be utilized to create more powerful antibacterial medications for the management of prevalent illnesses caused by *Salmonella typhi*, *P. aeruginosa*, and *Escherichia coli*. The text

**Table 4: The zones of inhibition for the ligand and its complexes**

<i>Bacteria</i>	<i>E. coli</i>			<i>P. aeruginosa</i>			<i>S. typhi</i>		
<b>Con.</b> <i>μg/mL.</i>	500	250	125	500	250	125	500	250	125
<b>L</b>	15	12	9	10	7	-	11	8	6
<b>1</b>	16	14	10	8	-	-	9	6	-
<b>2</b>	12	10	9	11	8	6	17	12	10
<b>3</b>	8	6	-	7	-	-	10	9	6
<b>4</b>	11	9	8	11	6	-	11	8	6
<b>AMC</b>	13			12			13		
<b>GEN</b>	12			12			12		

(AMC)= Amoxyclav, and (GEN)= Gentamicin



**Fig. 5: inhibition zones of complexes**

## 11. Conclusions

It was accomplished to synthesize and analyze the Schiff base ligand 4,4'-(ethane-1,2-diyl)bis(N-(pyridin-2-ylmethylene)aniline) (C<sub>26</sub>H<sub>22</sub>N<sub>4</sub>), as well as its complexes, formula

[M(L)(H<sub>2</sub>O)<sub>2</sub>](OAc)<sub>2</sub> (M = Mn (II), Zn (II), the formula [M(L)Cl<sub>2</sub> for Pd (II) and Ag (I) complexes. A stable five-membered chelate ring was formed when the ligand linked to the metal ion through the nitrogen of pyridine and azomethine. An octahedral and square planer shape has been suggested for the metal complexes. Under the same experimental settings, the complexes showed superior antibacterial activities than the parent Schiff base ligand. This study also suggests that as concentrations of the produced chemicals increased, so did their capacity to inhibit bacterial growth.

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